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Fabrication and Certification of Electroformed Microhardness Standards

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ABSTRACT: A commonly quoted characteristic of a material is its hardness. Accurate measurements of this characteristic require the use of precise standards to verify that the testing instrument, procedures, and comparisons with the results of another laboratory are accurate; however, it is recognized that currently available standards vary considerably in hardness from point to point across the surface. Electroplating technology was utilized to fabricate new microhardness standards. This technology provides a means for obtaining uniform hardness by close control of process variables which determine grain structure and composition.

Two microhardness standards are now in production—one with a hardness of ~ 125 kg/mm^2 and the other at ~ 600 kg/mm^2. The hardness values are certified at loads of 0.245, 0.490, and 0.981 N (25, 50, and 100 gf) with both Vickers and Knoop indenters. These electroplated materials have standard deviations in hardness, particularly at low loads, that are significantly better than the available standards. The fabrication of the new standards, their certification procedures, and testing instrument characteristics are discussed.

KEY WORDS: hardness, Knoop hardness, Vickers hardness, hardness testing metrology, nondestructive testing, testing instruments, microindentation hardness testing

Microhardness standards serve as an important means of quality control, not only for electrodeposited coatings, but also for other applications, and they can be used to ensure that the testing instruments are operating properly. Presently, the only standards available are produced by the makers of the testing instruments, and these not only lack a uniform standard for certi-

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fication of hardness but also exhibit significant variation of hardness across their surfaces.

At present, all commercial standards are produced from alloys prepared from a metallurgical cast process. The main drawback with this process is the difficulty in controlling cooling of the alloy melt. Failure to cool evenly produces a variable grain structure and composition. Electrodeposition methods of fabricating microhardness standards which do not have these drawbacks have been developed at the National Bureau of Standards (NBS). By very closely controlling current density, temperature, and electrolyte agitation, it is possible to produce extremely uniform electrodeposited material, which results in less variability in hardness from point to point across the surface. To date, two standards have been produced, one from a "bright copper" electrolyte and another from a "bright nickel" electrolyte. Electroplated bright copper was chosen because its nominal hardness of 125 kg/mm^2 is not only widely used but closely represents the hardness of some commonly used noble metals. Electrodeposited bright nickel was chosen because its nominal hardness of 600 kg/mm^2 closely represents the hardness of some commonly used ferrous metals.

Electroplating, using proprietary brighteners, produces grain refinement and grain distribution superior to that achieved with no brighteners, resulting in significantly more uniform hardness, as well as bright, smooth surfaces even before any polishing. Proprietary brighteners contain a leveling agent, usually an organic, absorbed on the surface of the high current density areas or peaks on the plating surface. This organic may inhibit plating on these high current density areas but allows plating to continue in the valleys, thereby providing a leveling effect. Another characteristic of the bright plating electrolytes is very good micro-throwing power, that is, the ability to deposit metal in grooves and cracks where these surface imperfections are of a microscopic nature.

Experimental Procedures

Fabrication Procedure

The electrolytes used in the electroplating of microhardness standards are commercially available copper and nickel solutions. The anodes were 0.00490% phosphorized copper and low-sulfur, low-cobalt nickel bars placed in anode bags. The power supply was a 15-A, 100-V constant-current source. The electrolyte agitation was provided by pumping filtered air through a sparger [perforated tube of polyvinyl chloride (PVC)]. The substrate used in this process was a 22.5 by 45-cm sheet of polished AISI 1010 steel, mounted in a Teflon box. Deposits of 500 μm of copper on a copper substrate and on a steel substrate showed no significant difference in hardness, therefore, the less expensive steel substrate was chosen. Mounting the substrate in a box with the open side facing the anodes in the electrolyte provides for a much more uniform current distribution. A uniform current density is necessary for uniform grain size, which is essential for this project. Electroplating is complete when 1 mm of copper or nickel is deposited.

After the copper or nickel has been deposited, a 2.5-cm strip is removed from all four sides of the plate to ensure uniformity of thickness across the plate. The plate is then cut into 1.35-cm-square specimens and placed in a stainless steel ring, 2.5 cm in outside diameter and 1.0 cm in height. The stainless steel ring was polished to ensure coplanar surfaces. This ring is then filled with an epoxy medium, used as a mold to enhance uniform polishing. The mold is polished on an automatic system. The polishing procedure removes approximately 125 μm of material from the original coating of 1 mm, thus leaving a substantial coating thickness to prevent the steel substrate from having any effect on the hardness measurement. The resultant coating thickness allows for a ratio of a minimum of 100:1 for coating thickness to depth of indentation when the hardness is determined at the maximum certified load of 0.981 N (100 gf).

Certification Procedure

The certification of these test blocks was made in accordance with the ASTM Test for Microhardness of Electroplated Coatings (B 578-80) and Test for Microhardness of Materials (E 384-84). The test instrument load-time response was measured using a compression load cell, calibrated with NBS-certified weights, to ensure conformity to ASTM Test E 384, Part B. The load cell was used to determine the actual load being applied to the test block during the time of indentation, as well as to determine the dwell time for full-load application. The optical measuring system of the test instrument was calibrated by an NBS-certified stage micrometer. The hardness indentations were made in all four corners and in the center of the test blocks and were measured using a $\times 100$ objective lens having a numerical aperture of 0.90. The hardness values are certified at loads of 0.245, 0.490, and 0.981 N (25, 50, and 100 gf). Vickers and Knoop indentations are made on separate specimens.

The first step in the certification of the microhardness standards was to identify and investigate sources of error which influence the accuracy of the certified values. The identifiable sources of error are (1) indentation measurement due to the optics or operator variability, (2) test instrument loading, and (3) test block variation.

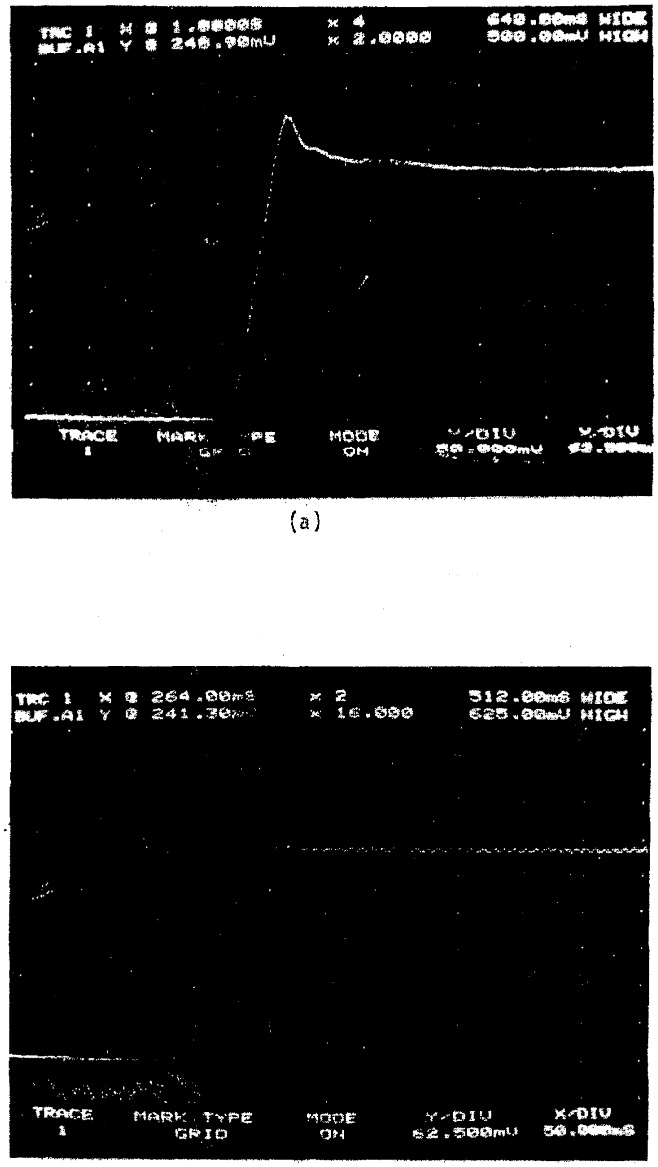
It is generally acknowledged that the greatest source of error is the optical measurement. The first step that was taken to reduce this error was to use an optical system with a $\times 100$ objective with a numerical aperture of 0.90, thus achieving a total magnification of 1000 with better resolution than is normally found on most test instruments, in which the objective power is typically $\times 40$

or ×50 and the numerical aperture is 0.65. The tips or ends of the microindentations were more readily defined, resulting from the use of the higher magnification and resolving power. In using this optical system, each operator determined the calibration factor using a certified stage micrometer. This calibration factor was verified each time a test block was measured. It was also found that better reproducibility of a measurement was obtained when using a filar micrometer eyepiece, which incorporated a double line just below the single cursor line. The tips of the microindentation are centered between the double lines and referenced to the single line just above the tip of the microindentations. The incorporation of all the previously mentioned steps has greatly reduced the "scatter" of the measurements.

The second source of error that was investigated was test instrument loading. A number of test instruments were evaluated, and it was discovered that impact loading, ringing (oscillations) resulting from impact loading, and undamped vibration were associated with the various test instruments, especially at loads of 0.245 N (25 gf) and less.

A miniature precision load cell was used to determine the actual load being applied during the time of indentation. The load cell was calibrated with certified weights, and an indentation was then made directly on the load cell. The load cell featured a peak/hold option which recorded the peak load applied during the test. The load-time responses of the test instruments were recorded on a digital oscilloscope. One instrument indicated a 15 to 20% impact load above the preset load value, for example, a 10 gf load was actually measured to be 12 gf. Also associated with this impact load is a drop in load before a steady-state preset load value is reached. A second instrument gave an impact load 20% greater than the preset load, which resulted in a long ring down time before a steady load value was reached. The load-time response of a third test instrument showed smooth uniform load application with an impact load of 20% greater than the preset value but with little ring down. None of these three instruments incorporated any damping mechanism to prevent impact loading or ringing. A fourth instrument was evaluated, and the associated load-time response showed no impact loading. This instrument was hydraulically damped. An example of a load-time response from an instrument which produces an impact "overshoot," is shown in Fig. 1*a*, along with a load-time characteristic from an instrument which applies the load more gradually, in Fig. 1*b*. The microhardness standards are presently being indented on a hydraulically damped instrument in which the actual load is within 0.5% or less of the applied load. Although impact loading may be large only at low applied loads, this error becomes significant when producing NBS-certified standards.

The third source of error investigated was variation in the hardness of the test block resulting from the grain size or composition variation. The variation in hardness across the test block is illustrated in Fig. 2, which represents hardness impressions on NBS standards compared with two different com-



(a)

(b)

FIG. 1—*Plots of load-time response for microhardness test instruments showing* (a) *20% impact loading above the preset value of 10 gf resulting from an undamped test instrument and* (b) *no-impact loading above the preset value of 10 gf from a hydraulically damped instrument: 1.0 gf = 33.33 mV; x axis = time, 62.5 ms/cm; y axis = load, 50 mV/cm.*

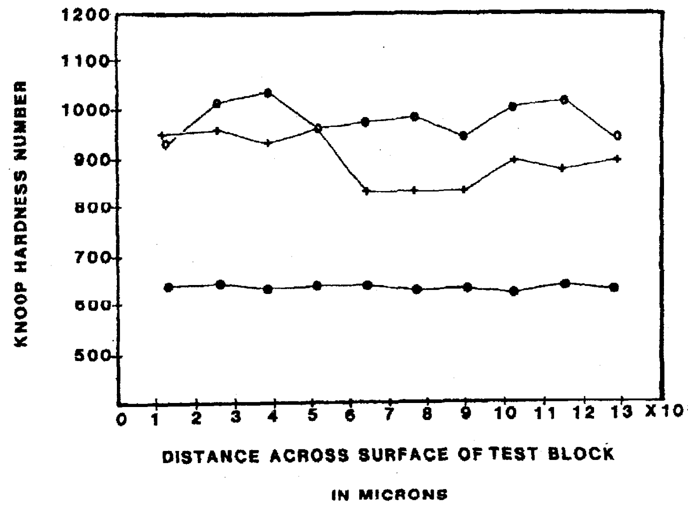


FIG. 2—*Comparison of the NBS nickel microhardness standard with two commercially available steel test blocks illustrating the variation in hardness across the test surfaces when using a 25-gf load: (•), NBS nickel standard; (○) commercial steel Test Block A, (+) commercial steel Test Block B.*

mercial steel test blocks. An impression was made every 1250 μm across the surface, and the hardness was plotted as a function of distance. The load used was 25 gf.

Results and Discussion

The results of a comparison of measurements of the same indentation using an optical system with an objective with a numerical aperture of 0.65 and a total magnification of ×400 (×40 objective and ×10 eyepiece) and an optical system using an objective with a numerical aperture of 0.90 and a total magnification of ×1000 (×100 objective and ×10 eyepiece) are tabulated in Table 1. One indentation at each of three values of load was made at five positions on the test blocks. The average hardness value and standard deviation in hardness units are reported for each set of five indentations for each load value. Each of these indentations was measured using both optical systems. The lower values of hardness and smaller standard deviations were obtained with the use of the optical system that incorporated the higher numerical aperture objectives and higher magnifications. This objective numerical aperture effect was also demonstrated by Tarasov and Thibault [1] and Brown and Iveson [2]. Higher resolution and magnification increased the ability to determine the ends of the indentations, resulting in greater diagonal lengths and less variation.

The use of a precision load cell to determine the load applied during the time of indentation resulted in the discovery of impact loading, oscillations

TABLE 1—*Optical measurement: comparisons of ×400 and ×1000 objectives.*

Nickel				Copper			
Load Value, g	Position	Instrument A, ×400	Instrument B, ×1000	Load Value, g	Position	Instrument A, ×400	Instrument B, ×1000
			KNOOP HARDNESS				
25	1	721.7	627.9	25	1	133.6	124.7
	2	702.6	644.1		2	128.5	124.2
	3	690.3	633.7		3	135.1	126.6
	4	696.4	658.0		4	138.3	126.6
	5	721.7	666.6		5	131.5	126.6
	Average	706.5	655.8		Average	133.3	126.0
	SD	14.5	10.0		SD	4.3	1.6
50	1	730.8	622.7	50	1	131.3	128.5
	2	735.5	637.7		2	130.6	127.8
	3	661.2	641.5		3	130.2	127.1
	4	699.1	637.7		4	129.2	126.1
	5	703.5	626.4		5	137.2	129.9
	Average	706.0	633.2		Average	131.7	127.8
	SD	29.8	8.2		SD	3.2	1.4
100	1	690.3	627.9	100	1	129.3	128.3
	2	660.8	625.3		2	132.5	128.3
	3	715.3	633.3		3	135.4	129.0
	4	666.6	635.9		4	131.8	132.3
	5	663.7	633.3		5	131.3	127.1
	Average	679.3	631.9		Average	132.0	129.0
	SD	33.3	4.6		SD	2.2	2.0
			VICKERS HARDNESS				
25	1	724.3	578.7	25	1	114.1	115.3
	2	626.8	612.4		2	115.8	112.4
	3	689.4	585.2		3	115.3	111.9
	4	641.6	612.4		4	121.9	114.7
	5	752.3	619.5		5	129.0	112.4
	Average	697.7	601.6		Average	119.2	113.3
	SD	54.6	18.4		SD	6.2	1.5
50	1	683.1	612.8	50	1	123.4	116.5
	2	695.0	633.2		2	118.6	115.3
	3	683.1	617.8		3	120.8	115.7
	4	649.2	617.8		4	126.2	119.1
	5	689.0	643.8		5	128.1	115.7
	Average	679.8	625.0		Average	123.4	116.4
	SD	17.8	13.0		SD	3.9	1.5
100	1	672.9	645.4	100	1	124.4	115.8
	2	626.8	653.1		2	124.4	118.2
	3	664.9	630.4		3	121.2	115.6
	4	664.9	660.9		4	128.7	117.0
	5	689.4	645.4		5	126.0	115.8
	Average	663.7	647.0		Average	124.9	116.4
	SD	23.0	11.3		SD	2.7	1.1

TABLE 2—*Operator comparison of Knoop hardness across test block by optical measurement using a ×100 objective.*

		Load Values					
		25 g		50 g		100 g	
Position	Indentation	Operator A	Operator B	Operator A	Operator B	Operator A	Operator B
1	1	605.4	605.4	617.7	627.9	612.4	615.9
	2	612.4	612.4	617.7	612.7	619.5	626.7
	3	612.4	598.5	617.7	612.7	612.4	615.9
	4	591.8	598.5	622.8	612.7	615.9	615.9
	5	598.5	591.8	607.8	612.7	612.4	619.5
	Average	604.1	601.3	616.8	615.8	614.5	618.8
	SD	9.0	7.8	5.5	6.8	3.2	4.7
2	1	598.5	591.8	617.7	622.8	608.9	605.4
	2	612.4	585.2	612.7	622.8	612.4	612.4
	3	598.5	585.2	617.7	612.7	608.9	608.9
	4	605.4	585.2	607.8	607.8	612.4	601.9
	5	598.5	591.8	612.7	612.7	608.9	605.4
	Average	602.7	587.8	613.7	615.8	610.3	606.8
	SD	6.2	3.6	4.2	6.7	1.9	4.0
3	1	598.5	605.4	612.7	607.8	612.4	615.9
	2	598.5	585.2	617.7	617.7	605.4	605.4
	3	591.8	598.5	617.7	607.8	612.4	619.5
	4	598.5	598.5	612.7	607.8	608.9	605.4
	5	591.8	598.5	612.7	602.9	608.9	612.4
	Average	598.8	597.2	614.7	608.8	609.6	611.7
	SD	3.7	7.4	2.7	5.4	2.9	6.3
4	1	591.8	605.4	602.9	622.8	615.9	605.4
	2	591.8	591.8	612.7	607.8	608.9	612.4
	3	591.8	598.5	612.7	612.7	612.4	608.9
	4	598.5	605.4	598.1	612.7	612.4	623.1
	5	591.8	598.5	612.7	622.8	612.4	608.9
	Average	593.1	599.9	607.8	615.8	612.4	611.7
	SD	3.0	5.7	6.9	6.7	2.5	6.8
5	1	598.5	598.5	617.7	622.8	619.5	612.4
	2	598.5	598.5	598.1	607.8	612.4	615.9
	3	578.6	585.2	617.7	622.8	615.9	612.4
	4	585.2	578.6	612.7	617.7	612.4	612.4
	5	591.8	612.4	622.8	617.7	608.9	608.9
	Average	590.5	594.6	613.8	617.8	613.8	612.4
	SD	8.7	13.1	9.5	6.1	4.0	2.5
All indents							
	Average	597.2	596.2	613.4	614.8	612.1	612.3
	SD	8.1	8.9	6.4	6.6	3.4	6.1

resulting from impact loading, and undamped vibrations. With regard to the effect of vibrations on microhardness testing, the results of this work are in agreement with those of prior work by Campbell [*3*].

Once the errors associated with the optical measuring system and test instrument loading were addressed and reduced, operator variability was investigated. An NBS bright nickel standard was used for this portion of the study. Five indentations at three values of load were made at five positions on the test block. Two operators (A and B) measured the same indentations with very similar results in average hardness values and standard deviation for the 25 indentations made at each load. These results are shown in Table 2 for each set of indentations at different positions for the three load values. The difference in the average hardness value determined by each operator for the 25 indentations at each load was a maximum of 1.4 hardness units. The improved optical measuring system greatly reduced the variability between operators.

The comparative results of multiple indentations on NBS standards and commercial available test blocks are shown in Table 3. The lower standard deviation obtained for the NBS standards is indicative of small variations in hardness from point to point across the test surface, whereas, the standard deviations for the commercial test blocks were much larger because of variation in grain sizes or material composition (different phases).

Conclusions

Errors resulting from optical measurement can be reduced by using high numerical aperture objectives.

Many commercial microhardness test instruments were found to introduce errors from impact loading and internal vibrations when used at low loads of 25 gf and less.

Many commercial standards have nonuniform microstructures, resulting in point to point variation in the hardness value.

Electrodeposited materials have a much more uniform microstructure.

Microhardness standards prepared by electrodeposition technology are significantly better than commercially available test blocks when used for low load testing of 200 gf and less.

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